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# Effect of Commercial Titanium Dioxide on the Properties of Mortars Cured in Different Environments

**Assem A. Aliem[1], Hanaa I. El-Sayad[2], Adel E. El-Ghaly[3], Sahar Moussa[4]**

[1]Professor of Properties and Testing of Materials, Civil Engineering Department, Faculty of Engineering at Shoubra , Benha University
[2]Professor of Properties and Testing of Materials, Civil Engineering Department, Faculty of Engineering at Shoubra, Benha University
[3]Lecturer, Civil Engineering Department, Faculty of Engineering at Shoubra, Benha University
[4] PhD Candidate- Housing and Building National Research Center

## ملخص البحث :

تستخدم مادة نانو ثانى اكسيد التيتانيوم فى انتاج خرسانة تعمل على تحسين الخواص الميكانيكية ولكنها تسبب مشاكل كبيرة تتعلق بتكلفة الصناعة وصحة الانسان. هذه الدراسة تبحث فى إمكانية إستخدام بودرة ثانى اكسيد التيتانيوم التجارية كمادة مضافة الى المونة بنسب مختلفة. ودراسة تاثير بيئات مختلفة لمعالجة المونة من ناحية نسبة الماء الممتص بعد سبعة ايام من المعالجة فى الماء و نسبة الفقد فى الوزن نتيجة فقد الماء بالتبخر للعينات فى الهواء والعينات فى غرفة ثانى اكسيد الكربون وكذلك التعرض لهجوم الكبريتات. وقد أوضحت النتائج قلة نسبة الماء الممتص مع زيادة نسبة ثانى أكسيد التيتانيوم وزيادة فى الفقد فى الوزن نتيجة تبخر الماء. ولم يظهر اثر للشروخ فى للعينات المغمورة فى محلول تركيزه 5% من كبريتات الصوديوم.

## 1. ABSTRACT

Nano-particles can be used in cement-based materials to improve mechanical properties, however, their utilization may cause great problems related to cost and human health. This paper investigates the possibility to use commercial grade $TiO_2$ as an additive to mortar with different percentages to realize some of the benefits of $TiO_2$. The research explores the effect of different environments on absorbed water during 7 days curing, mass loss due to water evaporation of samples kept in air and $CO_2$ chamber and resistance to sulfate attack. The results show a reduction in absorbed water as $TiO_2$ increased while the mass loss due to water evaporation increased. No cracks observed of samples fully immersed in 5% concentration sodium sulfate solution.

**Keywords**: $TiO_2$ Powder, Absorbed Water, Water Evaporation, Sulfate Resistance

## 2. INTRODUCTION

The interest in use of titanium dioxide in construction materials stemmed initially from its white color and therefore for its suitability for a wide range of products including special architectural effects. However, the recognition that certain forms of titanium dioxide have photocatalytic properties has widened its application [1]. Heterogeneous photocatalysis was first discovered by Fujishima and Honda in the 1970's. It is a process involving a catalyst that absorbs UV energy from the sun and oxidizes or decomposes organic matters in either the atmosphere or aquatic environments [2].

In addition to photocatalytic properties, it is chemically and biologically inert, non-toxic, which makes it an accessible material for general applications. The study of the usage of $TiO_2$ in construction materials as a photocatalytic material initiated from the early 1990s [3]. Nano-engineering, or nano modification, of cement-based materials implies adding nano-size cement additives during mixing, to enhance and control some of the properties, including hydration, mechanical performance, and degradation resistance. Nano $TiO_2$ is known for its photocatalytic properties including $NO_x$

oxidation, removal of volatile organic compounds and self-cleaning. Due to these novel functionalities, it has been used with construction materials especially those exposed to high levels of pollution. The applications of nano $TiO_2$ include self-cleaning, air and water purification, self-sterilizing, and anti-fogging surfaces [4].

Prior research has examined the photocatalytic properties of nano $TiO_2$ itself [4, 5], as well as $TiO_2$ containing cement-based materials [3, 6]. The majority of this effort has been on characterizing and enhancing the photocatalytic efficiency.

A number of research studies have suggested the use of a thin film application of powdered $TiO_2$ as a coating to a number of substrates [7]. The number of titanium dioxide patents is continually growing and currently include materials in concrete tiles, concrete paving, white cement (architectural concrete), on building surfaces, as well as applying environmentally-friendly cement (TioCem) [8,9].

An investigation was conducted to study the effects of $TiO_2$ on the physical and mechanical properties of concrete containing 45% ground granulated blast furnace slag (GGBFS) as a binder. The results showed that the addition of up to 3% $TiO_2$ nano particles by mass could increase the flexural strength, improve the pore structure and dispersion of the particles, and progress the formation of hydrated products [10].

Introducing nano titanium dioxide into cement mortars would lead to a considerable increase in early age compressive strength and the higher nano $TiO_2$ dosage, the greater the improvement. However, it had an adverse effect on the later age strength [11]. The total porosity of $TiO_2$ blended pastes was decreased and the reduction of pore volume occurred mainly within the capillary pore range. The acceleration of hydration rate and the change in microstructure also affected the physical and mechanical properties of the cementitious materials. The smaller the nano $TiO_2$ particles resulted in higher water demand and shorter setting time and the compressive strength of the mortars was significantly improved, practically at early ages [12].

Using of $TiO_2$ nano particles as filler or replacing part of cement, is improving the performances of self-cleaning concretes. The compressive strength increases with increasing the amount of $TiO_2$ nano particles up to 1%. This is because pozzolanic activity of $TiO_2$ in hydrating cement attributed to the improvement of compressive strength of the modified concrete [13].

The commercial grade $TiO_2$ can also be used on the outer layers of building materials, like plaster or decorative cement based paints, to produce an economic and health hazard free layer, having the ability to reduce urban pollution [14]

A research was conducted to clarify the basic properties of mortar and concrete using $TiO_2$ as an admixture; on the fresh properties, strength properties, drying shrinkage, and carbonation depth. By replacing fine aggregates and cement with different $TiO_2$ percentages, they found that as the aggregate replacement amount of $TiO_2$ increases, the compressive and flexural strength also increased [15].

An investigation was established to study, using micro-sized $TiO_2$ powders, the photodegradation of pollutants $NO_x$ and toluene. The results showed that for all micrometric $TiO_2$ powders, even though they are sold as not photocatalytic materials, showed good results compared with the nano $TiO_2$ powders. Therfore, it was recommended to have the attention to the use of micrometric $TiO_2$ powders with the aim to reduce health problems associated to the difficult recovery and consequently to the inhalation typical of nanoparticles. In addition, the evaluation of the photocatalytic reactions of micrometric commercial powders by changing different parameters and experimental conditions was also recommended [16].

It would be advantageous to realize the benefits associated with the use of commercial grade $TiO_2$ without the higher cost, health hazards of nano $TiO_2$ particularly respiration problems.

However, relatively little research was performed to assess the potential impact of the inclusion of commercial titanium dioxide on properties of cement based materials [14, 17]. Some of the properties incorporating commercial grade $TiO_2$ in building plaster need to be further investigated to fully understand its effects on such mortars. In this research, the effect of using $TiO_2$ with different percentage to enhance properties of mortar was investigated.

## 3. RESEARCH SIGNIFICANCE

The main purpose of this research was to investigate the properties of mortars containing commercial grade $TiO_2$ as an additive. The effect of $TiO_2$ on various mortar properties in different curing condition was tested.

## 4. EXPERIMENTAL PROGRAM

The experimental program has been developed to investigate the following:

1. The effect of $TiO_2$ content on absorbed water of mortar samples during 7 days of curing.
2. The effect of $TiO_2$ content on mass loss due to water evaporation from mortar samples kept in air, closed CO2 chamber and sulfate solution.
3. The effect of $TiO_2$ content on mass loss of mortar samples fully immersed in sulfate solution.

### 4.1. Materials

The Ordinary Portland Cement (OPC) used throughout the test program was Suez Cement (CEM I 42.5 N) having surface area of 3500 $cm^2/g$ and specific gravity of 3.15 $g/cm^3$ conforming to the requirements of ESS 4756-1/2013 [18]. The chemical composition of the cement is shown in Table (1). Titanium Dioxide ($TiO_2$) was in solid state (powder), having slight odor and white color. $TiO_2$ with high purity was used. The physical properties of $TiO_2$ as obtained from the manufacturer are illustrated in Table (2). Figure (1) for X-Ray Diffraction analysis of $TiO_2$ reveals that it is mainly anatase phase. Figure (2) shows the particle size distribution of $TiO_2$ powder. Locally available natural sand was used as fine aggregate.

**Table (1) Chemical Composition of Portland Cement**

| **Component** | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | CaO | MgO | $SO_3$ | $Na_2O$ | $K_2O$ | L.O.I |
|---|---|---|---|---|---|---|---|---|---|
| **Content (%)** | 20.39 | 5.6 | 3.43 | 63.07 | 2.91 | 0.7 | 0.38 | 0.35 | 2.06 |

**Table (2) Physical Properties of $TiO_2$**

| **Name** | **$TiO_2$ powder** |
|---|---|
| Particle size | 0.2912 - 0.6746 µm |
| Purity | 98 % |
| L.O.I | 0.13 % |

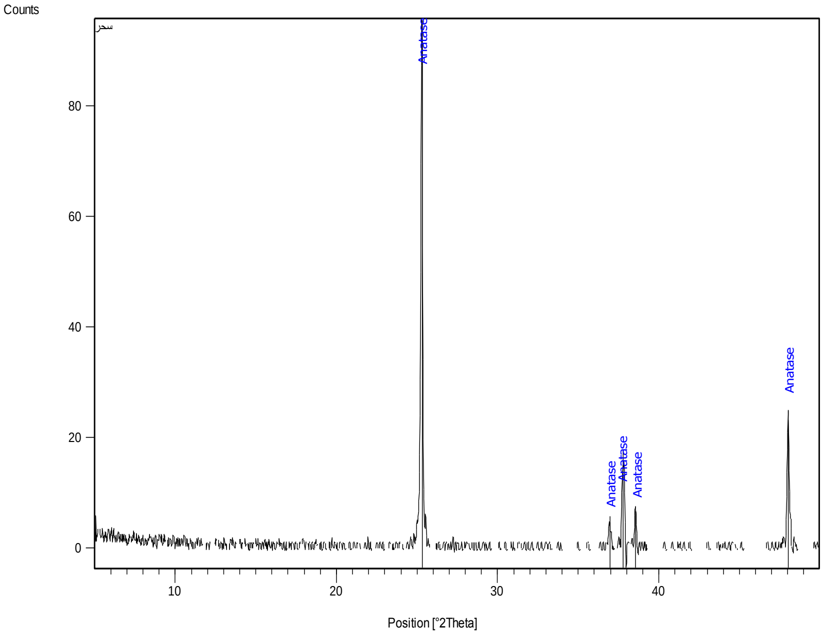
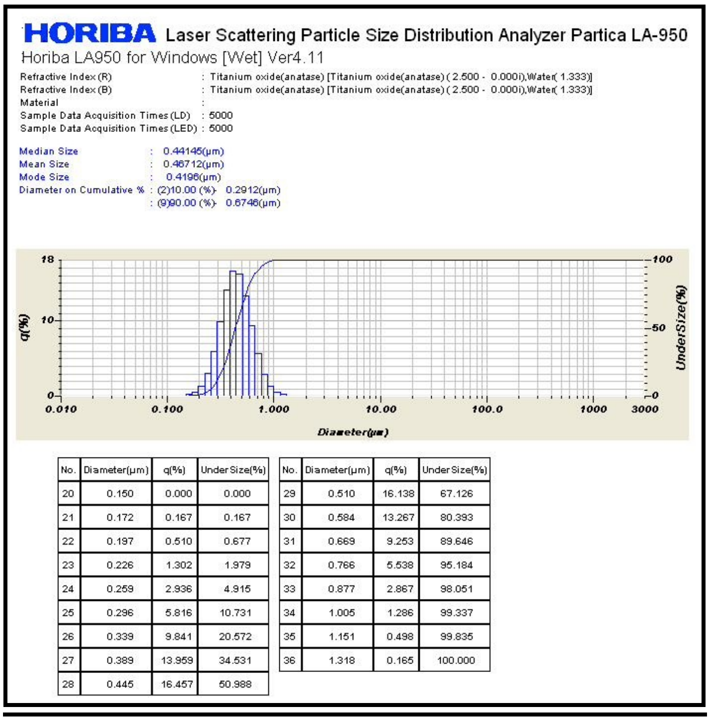


**Fig. 1 X-Ray Diffraction Analysis of $TiO_2$**


**HORIBA** Laser Scattering Particle Size Distribution Analyzer Partica LA-950

Horiba LA950 for Windows [Wet] Ver4.11

Refractive Index (R) : Titanium oxide(anatase) [Titanium oxide(anatase) ( 2.500 - 0.000i),Water( 1.333)]
Refractive Index (B) : Titanium oxide(anatase) [Titanium oxide(anatase) ( 2.500 - 0.000i),Water( 1.333)]
Material :
Sample Data Acquisition Times (LD) : 5000
Sample Data Acquisition Times (LED) : 5000

Median Size : 0.44145(μm)
Mean Size : 0.46712(μm)
Mode Size : 0.4196(μm)
Diameter on Cumulative % : (2)10.00 (%)- 0.2912(μm)
: (9)90.00 (%)- 0.6746(μm)

| No. | Diameter(μm) | q(%) | Under Size(%) |
|---|---|---|---|
| 20 | 0.150 | 0.000 | 0.000 |
| 21 | 0.172 | 0.167 | 0.167 |
| 22 | 0.197 | 0.510 | 0.677 |
| 23 | 0.226 | 1.302 | 1.979 |
| 24 | 0.259 | 2.936 | 4.915 |
| 25 | 0.296 | 5.816 | 10.731 |
| 26 | 0.339 | 9.841 | 20.572 |
| 27 | 0.389 | 13.959 | 34.531 |
| 28 | 0.445 | 16.457 | 50.988 |
| 29 | 0.510 | 16.138 | 67.126 |
| 30 | 0.584 | 13.267 | 80.393 |
| 31 | 0.669 | 9.253 | 89.646 |
| 32 | 0.766 | 5.538 | 95.184 |
| 33 | 0.877 | 2.867 | 98.051 |
| 34 | 1.005 | 1.286 | 99.337 |
| 35 | 1.151 | 0.498 | 99.835 |
| 36 | 1.318 | 0.165 | 100.000 |

**Fig. 2 Particle Size Distribution of $TiO_2$ Powder**

## 4.2 Mixes Description

A total of four mixes were prepared in the laboratory. The control mix was prepared from natural sand, cement and water. The water to binder ratio for all mortars mixes was set at 0.50. The cement content of all mixes was 350 kg/m$^3$. The cement to sand ratio for all mixes was set at 1:3. Other mixes were prepared with different contents of $TiO_2$ particles as an additive at 3%, 6% and 9% by weight of cement. An electrical mixer was used for mixing mortars. The samples were 50mm cubes and prisms 25mm × 25mm × 285 mm. The specimens were cast and compacted in two layers using a plastic compacting bar, where each layer was compacted 25 times. Then the molds were placed on compacting table and vibrated for about 15 seconds. The molds were immediately covered with plastic sheet to avoid moisture loss, and were kept at room temperature (23 ± 2°C) for 24 hours. The specimens were then demoulded and were kept in water for seven days for moist curing.

## 4.3 Testing Procedures

### 4.3.1 Absorbed Water

The absorbed water of each mortar specimens was determined after seven days of water curing. After demoulding the specimens, the weight was recorded. Then the specimens were kept in water for seven days. Then the specimens were taken out from water curing container and weight of each specimen was recorded.
The absorbed water was calculating as the following:

$$\text{Absorbed Water} = \frac{W_1 - W_0}{W_0} \times 100$$

Where:
$W_0$ = Weight of saturated specimen in air
$W_1$ = Weight of specimen after seven days of water curing
The absorbed water was calculated as an average for twenty seven cubes and for fifteen prisms for each tested mortar.

### 4.3.2 Mass Loss due to Water Evaporation

After 7 days of water curing, the specimens were placed in air for 90 days and weights were recorded at 28, 56, and 90 days. Mass loss was calculated for both cubes and prisms for each mortar. Mass loss was calculated as an average for three cubes and for five prisms for each tested mortar.

The percentage mass loss was calculating as the following:

$$\text{Mass Loss} = \frac{W_0 - W_1}{W_0} \times 100$$

Where:
$W_0$ = Weight of saturated specimen after seven days of water curing
$W_1$ = Oven dry to a constant weight at 105 °C of specimen at specified test age (28, 56, and 90 days)
Cubes and prisms were kept in $CO_2$ chamber for 90 days and the weight of specimens recorded at 28, 56, and 90 days respectively. Mass loss was calculated as an average for three cubes and for five prisms for each tested mortar. The details of the prepared cubes, prisms and tests performed are presented in Table 3.

### 4.3.3 Mass Loss due to Immersion in Sulfate Solution

The prisms were maintained in Sodium Sulfate solution of 5% concentration after seven days of water curing (five prisms for each mix). This test performed according to ASTM C1012 / C1012M – 15[19].The prisms were fully immersed in the solution kept in glass container. The prisms were kept at a distance of 50 mm away from the walls of the container. The container was covered in order to minimize the evaporation. The pH value was maintained neutral throughout the study period. Also the solution was stirred every week to avoid deposits on the base of the containers. The surface of the prisms was cleaned, weighed.  The weight of prisms was observed and recorded at ages 28, 56 and 90 days.

**Table (3) Details of Experimental Program**

| | **Test Program Table** | | | |
|---|---|---|---|---|
| | **Fresh State** | | | |
| **Tio$_2$ % added** | **Control (%0)** | **3%** | **6%** | **9%** |
| **Curing** | 7 days of Water Curing of Cubes and Prisms | | | |
| **Absorbed Water** | 24 Cubes and 15 Prisms for each mortar | | | |
| **Mass loss due to Water Evaporation** | 3 Cubes and 5 Prisms kept in Air for each mortar | | 3 Cubes and 5 Prisms kept in $CO_2$ Chamber for each mortar | |
| **Mass loss due to immersion in Sulfate Solution** | 5 Prisms for each mortar | | | |

# 5. EXPERIMENTAL RESULTS AND DISCUSSION

## 5.1 Absorbed Water

### 5.1.1 Cubes

Figure (3) shows the average of absorbed water for cube test samples after water curing for 7 days. It can be seen from Figure 3 that as $TiO_2$ percentage increased, the absorbed water decreased. This could be due to the fact that the $TiO_2$ particles acted as fillers, occupying space in the pore structure, but this improvement is not a result of formation of more hydration products [20].

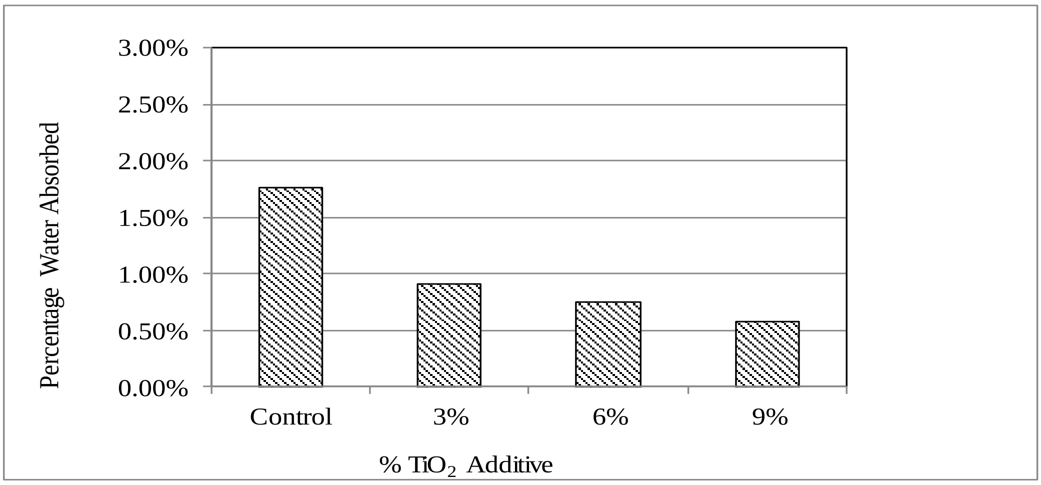


**Fig. 3 Absorbed Water of Cubes Cured for 7 days in Water**

### 5.1.2 Prisms

Figure (4) shows the absorbed water for the test prisms after water curing for 7 days. Figure (4) indicates that as $TiO_2$ percentage increased, the percentag**e** absorbed water decreased. The reason could be as the same reason discussed above for percentage absorbed water for cubes.

In a comparison of Nano $TiO_2$, it is clearly that grade $TiO_2$ has the same trend of water absorption property. As shown in Figure (5), the result could be related to the pozzolanic reaction which consumed $Ca(OH)_2$ creating more C–S–H this has led to a denser microstructure and consequently less water absorption. The reduction in water absorption with the increase of nano $TiO_2$ content in the mixtures resulted from enhancing the pore structure. Nano $TiO_2$ particles enhance the microstructure and causing a reduced porosity [21]. The degree of hydration at early hydration period was significantly enhanced by small dosages of nano-$TiO_2$ powder. Grade $TiO_2$ was confirmed to be non-reactive fine filler and had no pozzolanic activity. Also they acted as potential nucleation sites for the accumulation of hydration products [12].

An investigation of using nano silica (nano $SiO_2$) and nano titanium (nano $TiO_2$) in two types of self-compacting mortars (ratio binder: sand of 1:1 and1:2 with the same water/cement ratio and 30% of replacement of cement with fly ashes was established. The results showed that the water absorption by immersion increases with the addition both of nano-$SiO_2$ and nano-$TiO_2$, relative to the reference mortars (1:1 and 1:2). There is a slight decrease of porosity in the 1:2 family mixes relative to the 1:1 family mixes [26].

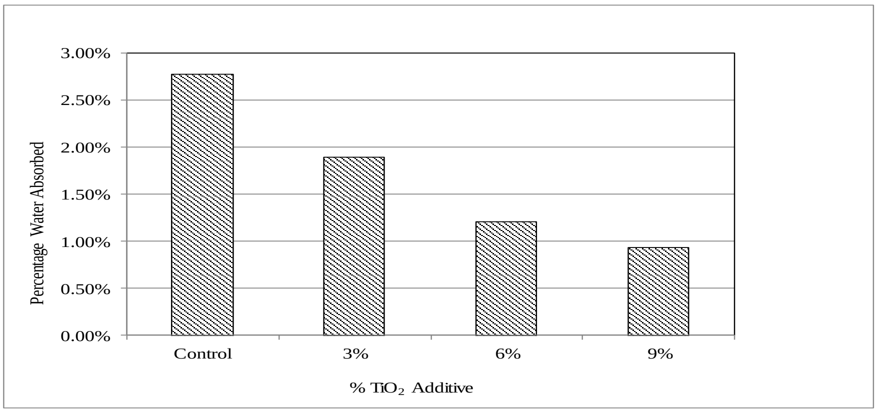


**Fig. 4 Absorbed Water of Prisms Cured for 7 days in Water**

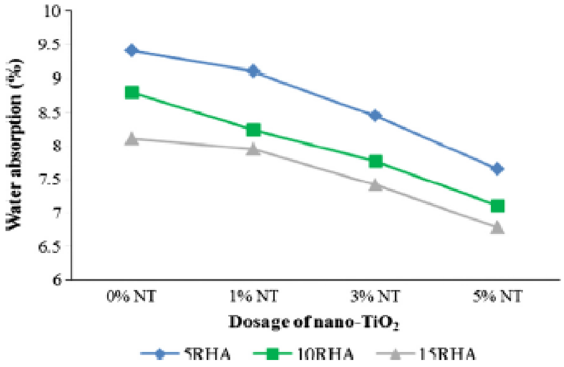


**Fig. 5 Water Absorption Results of Mortar [Mohseni et al., 2016]**

## 5.2 Mass Loss due to Water Evaporation

### 5.2.1 Cubes Kept in Air

Figure (6) shows the percentage of mass loss due to water evaporation for the test cubes kept in air at different ages. It can be seen that the percentage of mass loss is increasing by increasing $TiO_2$ percentage.

An experimental study illustrated that XRD diagrams of concrete samples containing nano-$TiO_2$ show peaks related to $Ca(OH)_2$ with approximately the same intensity as the normal concrete specimens [20]. This means that these particles do not react with $Ca(OH)_2$ in order to produce more hydration products [20]. This may explain the increase of percentage water evaporation with increasing of $TiO_2$ percentages. The finding from the test on water absorption up to 7 days and then water evaporation up to

90 days indicate that in mixes with $TiO_2$ there is less demand for curing water and more evaporable water at the end of curing.

The volume stability is a critical factor that governs the durability of cement-based materials, which is closely related to the water vapor transport features and the pore structure characteristics [22]. Influences of nano$TiO_2$ on the water loss of hardened cement paste samples due to water evaporation illustrated in Figure (7). It can be seen that the addition of nano-particles can reduce the water loss of the samples [22].

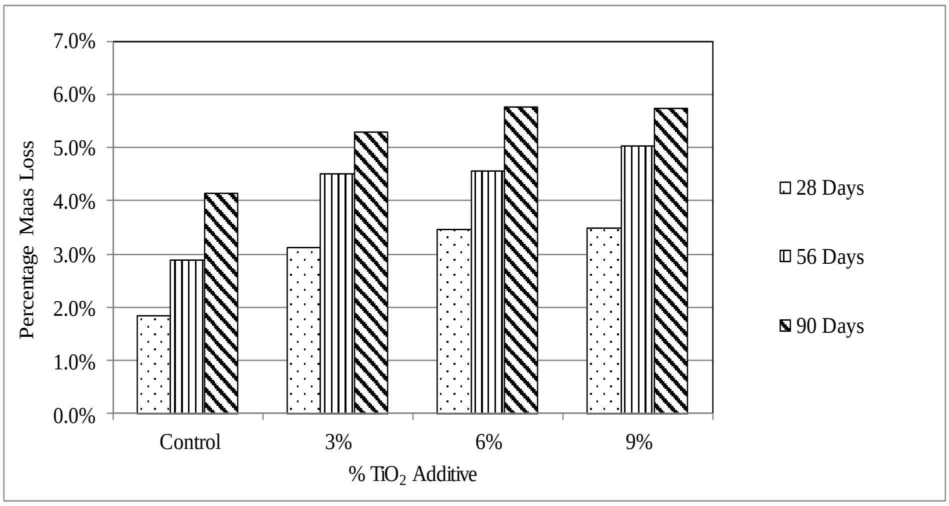


**Fig. 6 Mass Loss due to Water Evaporation of Cubes Kept in Air**

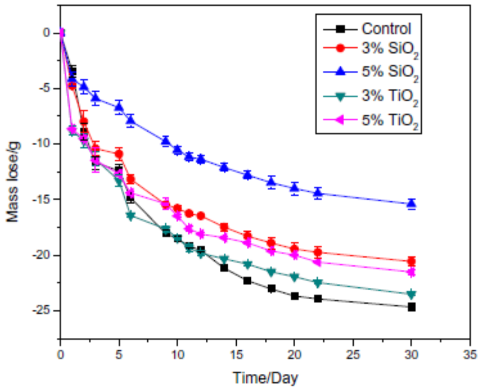


**Fig. 7 Influence of Nano Particles on the Water Loss of Cement Paste [Zhang et al., 2016]**

### 5.2.2 Cubes Kept in $CO_2$ Chamber

Figure (8) shows the percentage of mass loss due to water evaporation for samples stored in $CO_2$ chamber. It can be seen that the percentage of mass loss is increasing by increasing $TiO_2$ percentage. It also clear by comparing Figure (6) and Figure (8) that specimens kept in air lost more water compared to those stored in carbonation chamber. Storing the samples in $CO_2$ chamber means that they were in constant humidity environment and that explains the reduced water evaporation from those samples.

Figure (9) shows a photo of $CO_2$ chamber while the samples subject to $CO_2$, demonstrating the fog due to water vapor. Naturally, the water loss was less in the humid $CO_2$ chamber compared to that exhibited by the samples kept in laboratory air.

The difference is also evident in Figure (10). On average samples kept in $CO_2$ chamber lost less than 25% of those kept in air at 28 days. The decrease is also greater at later ages.

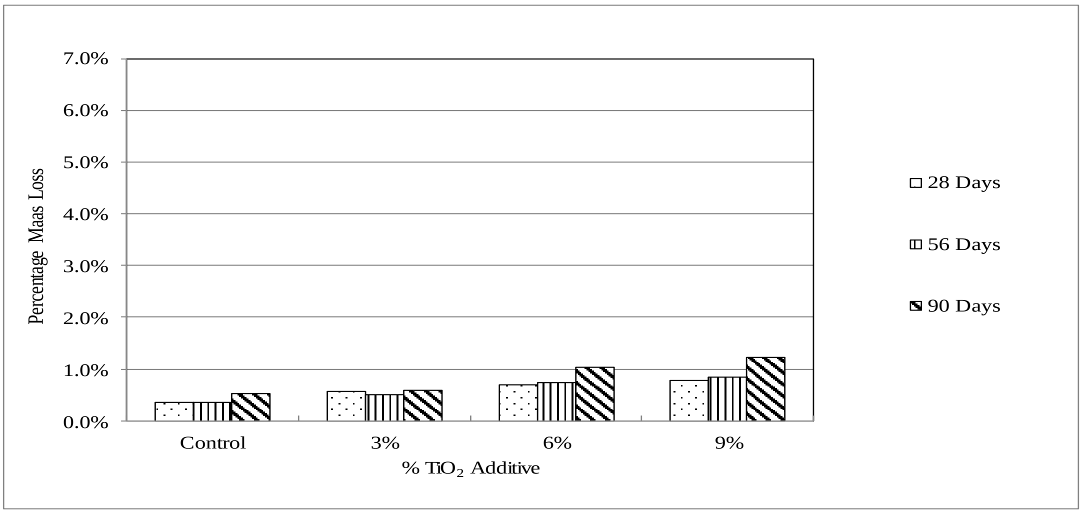


**Fig. 8 Mass Loss due to Water Evaporation of Cubes Kept in $CO_2$Chamber**

**Fig. 9 Humidity due to Water Evaporation in $CO_2$ Chamber**

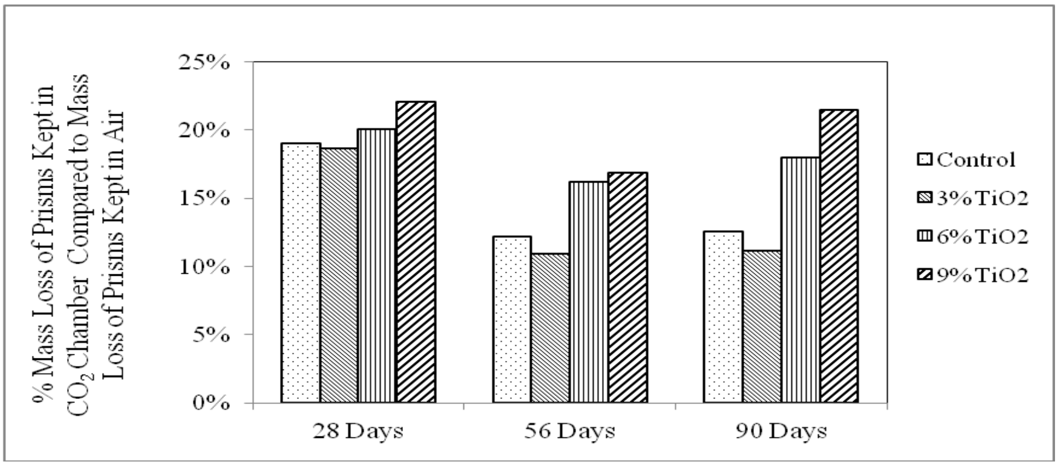


**Fig. 10 Mass Loss due to Water Evaporation from Cubes (Kept in $CO_2$ Chamber) Compared to Cubes (Kept in Air)**

## 5.3 Mass Loss of Prisms Kept in Sulfate Solution

Figure (11) shows test chamber for prisms immersed in 5% Sodium Sulfate Solution. Figure (12) shows the average of percentage mass change for samples kept in 5% concentration Sulfate solution ($Na_2SO_4$) for 90 days. It can be seen that the increase in mass of prisms is decreasing by increasing $TiO_2$ percentage at all ages. As shown in Figure (13), there was no cracking observed on the sample surfaces. The samples were cured for only 7 days and still exhibited reasonable sulfate resistance. The sulfate attack results are consistent with the water absorption results shown in Figure (4).

It seems that the inclusion of $TiO_2$ reduces the absorption of fluids into the mortar and has therefore enhanced the performance of samples in sulfate solution. Moist curing the concrete up to 28-days before exposure to physical sulfate attack was reported to reduce the damage due to sulfates of control OPC concrete specimens [23].

Referring to Figure (14), the samples of 5%, 10%, and 15% nano $TiO_2$ mortar prisms were immersed partially in 15% $Na_2SO_4$ solution developed heavy efflorescence within several days after the start of the experiment, with white deposits covering whole sample surfaces. However, no cracking was observed on the sample surfaces. This negatively impacts the aesthetics of a structure, but does not affect its soundness [24].

A previous research was conducted to study the effect of $Tio_2$ nano particles on Fly Ash concrete properties. The results showed highest resistance to sulfate attack due to addition of nano particles to Fly Ash concrete. Also Fly Ash-nano titanium dioxide specimens showed lesser weight loss compared to Fly Ash concrete without nano $TiO_2$ [25].

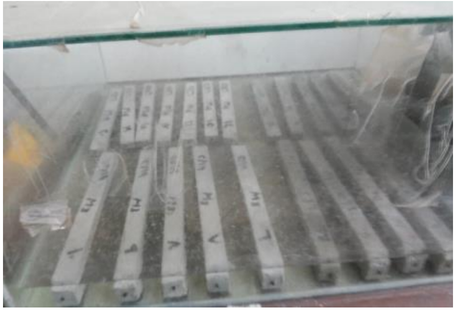


**Fig. 11 Prisms Immersed in 5% Sodium Sulfate Solution**

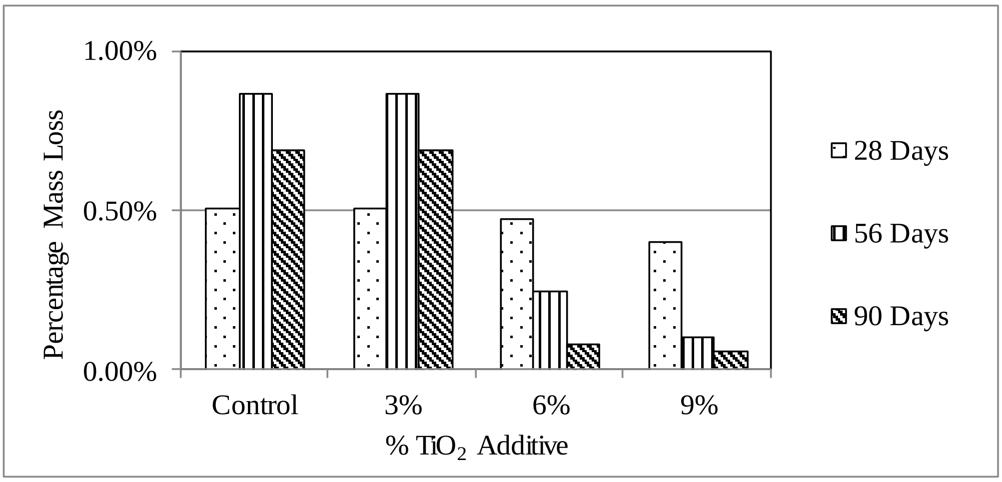
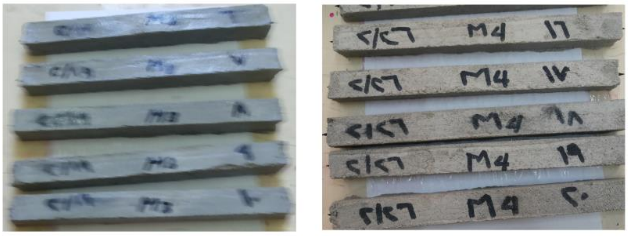


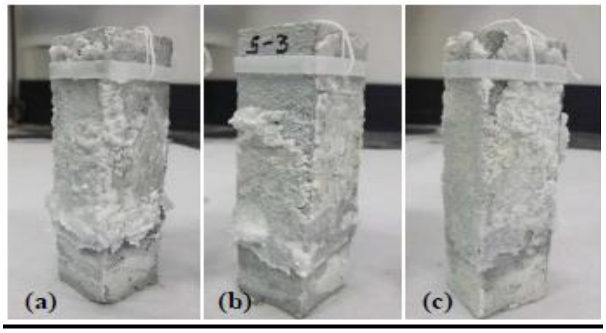
**Fig. 12 Mass Loss of Prisms Kept in Sulfate Solution Relative to Mass after 7 Days Curing**

Control 3 % $TiO_2$



6% $TiO_2$ 9% $TiO_2$

**Fig. 13 Appearance of Samples Fully Immersed in Sulfate Solution for 90 Days**



**(a) w/b=0.40 (b) w/b=0.50 (c) w/b=0.60**

**Fig. 14 Samples partially immersed in 15% $Na_2SO_4$ solution [Lee, 2012]**

## 6. CONCLUSIONS

1. As commercial grade $TiO_2$ percentage increased, the percentage absorbed water decreased. This indicates that increasing the percentage of $TiO_2$ in the mixture improves pore structure.
2. Mortars with $TiO_2$ cured in laboratory or $CO_2$ humid chamber for up to 90 days exhibited increased water loss compared to control samples without $TiO_2$.
3. The increase in mass of prisms immersed in sulfate solution is decreasing by increasing commercial grade $TiO_2$ percentage at all ages. No cracking was observed on the sample surfaces. Commercial grade $TiO_2$ mortar has good sulfate resistance at 5% concentration $Na_2SO_4$ solution. More severe environment with high concentration need to be investigated.
4. Referring to pervious investigation of nano $TiO_2$ at similar topic discussed in this research, it is recommended to investigate other properties of mortars containing commercial grade $TiO_2$. Comparing its performance to nano $TiO_2$ mortars with same percentages. Although the use of nano-materials in cementitious materials holds great advantages, problems related to lowering cost and human health still need to be resolved. In this research, using commercial grade $TiO_2$ in mortar has no adversely effect but need to be more investigated.

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